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Validation of a Method to Measure Polychlorinated Biphenyls in Natural Gas Pipelines

Grainger Labs., Inc.
Raleigh, NC

Prepared for

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Abstract

NIOSH-approved, commercially-available Florisil tubes have been found to quantitatively collect polychlorinated biphenyls (PCB) from natural gas streams when the sampling rate is less than 600 cc/min and when less than 230 liters of gas are collected. It has also been found that severe skewing of the Aroclor pattern can result when gas passes through a PCB contaminated pipe. When this occurs the pattern recognition approach usually used for quantifying PCB in air samples will be inadequate. In this situation confirmation should be done using a second GC column and, if polychlorinated biphenyls are confirmed, the Webb-McCall or the perchlorination procedure should be used for sample quantitation.

Background

In January 1981, polychlorinated biphenyl material (PCB or Aroclor) was found in condensate samples collected from a pipeline supplying natural gas to a small number of residences in New York. Air sampling in the kitchen of one of these residences showed very little difference in ambient air levels of PCB when the stove was operating or when it was shut off. Further, the levels found were below those found in many houses that have no natural gas sources (1). Subsequent analysis of condensate from pipeline drip traps showed the presence of PCB in a few other pipelines.

Unanswered were the questions, "Do PCB materials become entrained in the natural gas itself or do they stay in the condensate?" and "If they are entrained, is there a reliable way to collect them from a natural gas stream?" The experiments described below were done to answer these questions. The test method developed is contained in Appendix A.

Experimental and Results

Preliminary experiments showed that 2 g of Florisil doped with microgram quantities of Aroclor 1242 and contained in a 1.5 cm (OD) x 4.0-cm tube did not lose PCB when natural gas flowed through it at 1 ℓ/min for 15 hours. Florisil was selected for study because it is inexpensive, commercially-available in prepackaged tubes, easily cleaned and has been demonstrated to quantitatively collect PCB material in ambient air (2) and in stack samples (3). Because commercially-available National Institute of Occupational Safety and Health (NIOSH) approved sampling tubes contain only 150 mg Florisil, it was decided to also study the suitability of a larger tube for collecting PCB material, in case the smaller tube was found inadequate.

After demonstrating that Florisil did not lose PCB when natural gas flowed through it, a comparative study was done to determine if direct elution of PCB from Florisil using hexane would yield a cleaner sample than the desorption method in the NIOSH manual(2). In these experiments, two sets of NIOSH tubes were injected with amounts of Aroclor 1242, ranging from 1000 micrograms to 10 nanograms, and allowed to stand for 24 hours. One set was then eluted as follows: 1) the contents of each tube, including glass wool and polyurthane foam inserts, were removed and placed in a septum-capped vial; 2) 5.0 ml hexane was added, and (3) after two hours an aliquot was analyzed using a gas chromatograph (GC) equipped with an electron capture detector (EC). The other set of tubes was eluted as follows: 1) each tube was flushed with 2 ml hexane at a flow rate of 1 to 2 ml/min, beginning at the end opposite the injection point, 2) the hexane was collected in a calibrated vial and diluted to a total volume of 2.0 ml; and 3) the solution was analyzed using a gas chromatograph equipped with an electron capture detector (GC/EC).

As shown in Table 1, the elution procedure is superior to the desorption procedure, and thus, elution was used in all subsequent work. The elution technique also has the advantage that the sample volume can be easily reduced by evaporation to improve the detection limit. The most effective way to carry out elution is to place the contents of the tube into an extraction column where the hexane flow through the Florisil can be controlled to the desired rate of 1 to 2 ml/min.

The PCB capacity of the NIOSH-approved tube and that of the larger tube was then determined as a function of flow rate and concentration. In these studies, a modified gas chromatograph injector operated at 200°C was used to vaporize the Aroclor immediately upstream of the sampling tube. The vaporized Aroclor was carried to the tube by a natural gas stream. The results, summarized in Table 2 for the NIOSH tube and in Table 3 for the larger tube, showed that the tubes quantitively collected PCB when the amount of PCB injected was at least 100 µg. However, for unknown reasons, below 100 µg the collection efficiency of both types of tubes seemed to decrease as flow rate increased and PCB concentration decreased. Because the results were inconclusive and the experiments not representative of the real sampling situation, it was decided to continue to evaluate both tubes, but to do so under conditions more representative of the real sampling situation.

This latter study involved sampling a natural gas stream after it had passed through a U-shaped copper tube that had been contaminated by filling it with an Aroclor 1242 solution and then inverting the tube to allow the excess to drain. The copper tube was then connected to the system shown in Figure 1.

At the conclusion of each run, the apparatus was disassembled, the tubing between the Florisil tube and the isooctane cold trap rinsed with hexane,

and the rinsings added to the isooctane in the cold trap. The Florisil tube contents were placed in an extraction column and 5 ml of hexane was added. After 5 minutes the hexane was drained into a separatory funnel, and elution repeated. The hexane was then washed twice with concentrated sulfuric acid (to remove interferences) and once with distilled water and then passed through a sodium sulfate column into a Kuderna-Danish apparatus for concentration. The isooctane from the cold trap was washed and concentrated separately, but in an identical fashion. All extracts were analyzed by GC/EC.

Five sampling runs were carried out with this apparatus. The first two used the large tube and the last three the NIOSH tube. The first run was conducted before the copper tube was contaminated with Aroclor 1242 to serve as a blank. No peaks, observed in the chromatogram from either the Florisil or the isooctane, were attributable to Aroclor 1242 in this sample. The second and third runs used a copper tube that had been contaminated with a 10,000 ppm isooctane solution of Aroclor 1242. In each of these runs, the flow rate was 500 ml/minute for 100 minutes. The fourth run was conducted in a manner identical to the third except that flow rate was 500 ml/minute for 410 minutes. In the final run, a pipeline condensate containing about 20 ppm Aroclor 1242 was used rather than the isooctane solution.

As shown in Table 4, the capacity for PCB in even the NIOSH tube is excellent, so these tubes should be suitable for sampling natural gas streams. However, the analyst should be aware that a skewing of the Aroclor pattern may occur because of the different volatilities of the isomers in an Aroclor. For example, as shown in Table 5, the chromatogram for the Florisil sample is obviously skewed toward the more volatile isomers with respect to the isomer distribution in the original 10000 ppm Aroclor solution. If this situation is encountered when analyzing pipeline samples, the standard Aroclor-recognition

pattern method for identifying and quantifying the Aroclor present may be inadequate. In this situation the presence of PCB should be confirmed using a minimum of five peaks and two GC columns before any attempt at quantitation is made. If PCB is indicated, then either the Webb McCall(2,4) or the perchlorination(2,3) technique should be used for quantitation. The Webb McCall is the simpler procedure, since the weight of each isomer is directly determined using tables compiled by Webb and McCall and presented in their report. On the other hand, although the perchlorination procedure involves additional chemical treatment of the sample, it will definitely confirm if polychlorinated biphenyls were present in the original sample, something the Webb-McCall procedure cannot do.

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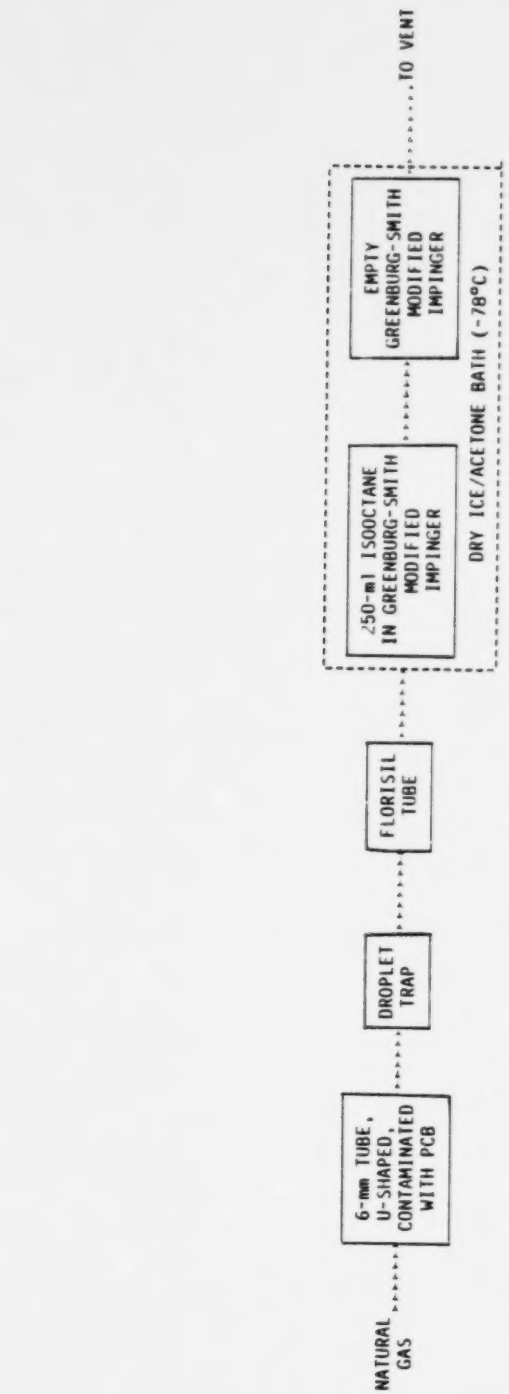


Figure 1. System used in contaminated pipe study

TABLE 1. RECOVERY OF AROCLOR 1242 FROM NIOSH TUBE

1242 μ	% Recovered	
	Desorption	Elution
1000	98.2	99.9
100	98.0	92.5
10	95.6	102
1	92.4	98.3
0.10	74.3	94.2
0.01	61.8	92.7

TABLE 2. COLLECTION EFFICIENCY OF NIOSH TUBE FOR AROCLOR 1242[a]

Flow (lpm)	% Recovery by sample weight			
	1000 μg	100 μg	10 μg	1 μg
100	98.6	95.9	75.3	ND[b]
0.50	99.6	100.2	90.4	62.8
0.20	ND[b]	99.1	92.3	75.7

[a]Florisil tube maintained near ambient temperature.

[b]Not determined.

TABLE 3. COLLECTION EFFICIENCY OF LARGE FLORISIL TUBE FOR AROCLOR 1242[a]

Flow (lpm)	% Recovery by sample weight				
	1000 µg	100 µg	10 µg	1 µg	0.1 µg
0.50	98.8	95.5	92.1	55.3	ND[b]
0.20	ND[b]	96.4	95.2	90.1	82.3

[a]Florisil tube maintained near ambient temperature.

[b]Not determined.

TABLE 4. EVAPORATION OF AROCLOR 1242 FROM A COATED-COPPER PIPE

Weight Florisil (g)	Type sample	Sampling time (min)	Sample volume (ℓ)	Aroclor 1242 (µg)		% On Tube
				Tube	Cold trap	
2	Blank	100	50	0	0	-
2	10,000 ppm	100	50	121	0.2	99.6
0.15	10,000 ppm	100	50	142	0.8	99.4
0.15	10,000 ppm	410	202	242	0.6	99.8
0.15	Condensate	240	170	11.5	<0.1	99.2

TABLE 5. COMPARISON OF ISOMER DISTRIBUTION IN AROCLOR 1242 BEFORE AND AFTER EVAPORATION IN A PIPELINE

Retention time (min)	% Peak area for each isomer	
	Orig. solution	Florisil tube
2.04	8.4	18.8
2.37	5.1	10.2
2.49	1.7	2.2
2.63	16.2	25.5
2.87	6.8	8.9
3.07	4.7	5.4
3.31	11.3	10.9
3.90	7.6	6.2
4.18	5.6	4.3
4.63	3.8	1.9
4.90	10.7	3.9
5.78	5.4	<1
7.68	1.5	<1
8.79	1.8	<1
9.66	7.8	<1

APPENDIX A

TEST METHOD FOR MEASURING POLYCHLORINATED BIPHENYLS IN NATURAL GAS STREAMS

1. Principle of the Method

A known volume of natural gas is drawn through a tube containing Florisil to adsorb PCB present in the gas sample. The collected PCB is desorbed with hexane, and the hexane is washed with concentrated sulfuric acid and analyzed using gas chromatography with electron capture detection (GC/EC). The concentration of PCB relative to a standard PCB preparation is then read from a standard curve. If the gas sample chromatogram differs significantly in composition from available PCB standards, then an alternate procedure such as perchlorination or the Webb-McCall calculation method is required for quantitation.

2. Range and Sensitivity

As written, the lower range of this method is estimated to be 2 $\mu g/m^3$ for a 50-liter sample. Concentrations as low as 0.1 $\mu g/m^3$ can be detected if the gas sample volume is increased, and the hexane extract volume is reduced by evaporation. The minimum detectable quantity of Arochlor 1242 was found to be 50 picograms per injection. No breakthrough was observed after sampling 200 liters of natural gas containing 1.2 mg/m^3 Aroclor 1242 at a flow rate of 500 cc/minute.

3. Interferences

3.1 Strict measures to avoid contamination are required when using the electron capture detector. The syringe must be thoroughly cleaned after each

injection. Hexane used for elution and back flush should be injected periodically for a purity check. It should show no peaks having retention times greater than 45 seconds, provided that not more than 5 microliters have been injected. To extend detector life, all samples should be cleaned with concentrated sulfuric acid before injection.

3.2 Any compound that causes a response in the election capture detector and has nearly the same retention time on the GC column as one of the PCB is an interference. This type of interference can often be overcome by changing the GC operating conditions or by selecting another column. Retention time data on a single column cannot be considered as conclusive proof of chemical identity unless the full Aroclor pattern is present. Sulfur-containing compounds in petroleum products that may interfere with the analysis are removed in the sample cleanup.

4. Apparatus

4.1 In rare cases where the natural gas line pressure is inadequate, a sampling pump may be required to obtain the desired 500 cc/min flow. If used, these pumps should be calibrated with a representative sorbent tube in the sampling line. A calibrated wet or dry test meter or a glass rotameter capable of measuring a flow rate of 500 cc/min within ± 5%, should be used for measuring sample flow and volume.

4.2 Sorbent Tubes. SKC Inc. No. 226-39 or equivalent. Each glass tube is at least 7 cm long with 4 mm i.d. and contains two sections of 30-48 or 30-60 mesh Florisil. The front section is preceded by glass wool and contains 100 mg Florisil, and the backup section contains another 50 mg. Urethane foam or glass wool is placed between the sections and after the backup section. The ends of the tube are flame sealed to prevent contamination before use.

4.3 Greenburg-Smith impinger, midget impinger packed with glass beads or equivalent to serve as knockout jar for condensate. Pyrex, stainless steel, Lexan are suitable materials of construction.

4.4 Valve. Micrometer to adjust sampling rate.

4.5 Orifice. Appropriately sized to reduce pressure when sampling high pressure lines.

4.6 Magnehelic or inclined manometer to measure line pressure.

4.7 Gas chromatograph equipped with an electron capture detector (^{63}Ni foil) capable of maintaining a detector temperature of 350° C.

4.8 Calibrated flow measuring device such as a bubble flow meter or a dry gas meter, capable of accurately measuring a 500 cc/min flow rate within ±2%.

4.9 Glass column (6 ft x 4 mm i.d.) packed with either a 1.5% OV-17/1.95% OV-210 on 100/120 mesh Supelcoport, a 4% SE-30/6% OV-210 on 100/120 mesh Supelcoport a 3% OV 210 on 100/120 mesh Supelcoport or any other suitable column.

4.10 Extraction columns

4.11 Microliter syringe, 10-micro liter, with chaney adapter.

4.12 Volumetric flasks, 10-ml, with glass stoppers.

4.13 A mechanical or electronic integrator or a recorder and some method for determining peak area.

4.14 Kuderna-Danish concentrators.

4.15 Volumetric Pipets, 4.0 ml Class A

5. Reagents

5.1 Hexane, pesticide quality.

5.2 Acetone, pesticide quality.

5.3 Sulfuric Acid, reagent grade.

5.4 Nitrogen, prepurified (carrier gas).

5.5 Standard PCB mixtures. Certified Aroclor 1242, 1254, and 1260 standards obtained from EPA or commercial suppliers.

5.6 Florisil--Floridin Company, 30/60 mesh, Grade A. The Florisil is cleaned by a procedure that includes an 8 hr Soxhlet extraction with hexane, drying for 8 hr in a 110°C oven, and activation by heating to 650°C for 2 hr (not to exceed 3 hr) in a muffle furnace. After allowing to cool to near 110°C the Florisil is transferred to a clean, hexane-washed glass jar, and sealed with a TFE®-lined lid. The Florisil should be stored at 110°C until it is placed in the tube and the tube sealed. Alternately, commercially available Florisil tubes such as SKC type 226-39 may be used as received.

5.7 p,p-DDE (1,1-Dichloro 2,2 bis[p chlorophenyl]ethene) for determining retention times.

6. Procedure

6.1 Cleaning of Equipment. Before use, all glassware used in the laboratory analysis and all equipment located upstream of the sampling tube should be washed in detergent and thoroughly rinsed with tap water, distilled water, pesticide-grade acetone, and pesticide-grade hexane. The glassware should then be dried.

6.2 Sampling.

6.2.1 General notes. The sampling techniques described below assume that a suitable valve is already installed on the sampling port to turn the flow on and off. The adapter required to connect the sampling equipment to the gas line is site dependent. However, one end of this adapter should have a Swagelok 0.25 inch (0.63 cm) tube fitting for attaching the SKC Florisil tube to the adapter. The tube can be mounted in a leak-free manner by placing

a 0.187 inch (4.7 mm) i.d. O-ring between the front and rear ferrules with the rear ferrule positioned opposite its normal direction, i.e., it should be facing the Swagelok cap. Before mounting the Florisil tube in the Swagelok fitting, each end of the tube should be broken to provide an opening at least one half the inside diameter of the tube. No rubber or Tygon tubing can be used upstream of the tube. The gas should be taken with the probe tip at least several inches from the inside wall to avoid contamination by condensate on the wall.

WARNING: Take extreme care when connecting and disconnecting the apparatus to the pipeline and when adjusting the flow rate and pressure. Before attempting to sample a pressurized pipeline, personnel must familiarize themselves with the behavior of natural gas as the pressure and flow rate are changed. The company operating the pipeline is likely the best readily-available source of this information and information on the location, condition and characteristics of the sampling valves.

During sampling, the Florisil tube should be mounted in the vertical position, if at all possible and the maximum gas pressure in the tube should be less than 20 cm H_2O. The sampling rate should not exceed 500 cc/min. The minimum volume should be 50 ℓ and the maximum should be 200 ℓ. A calibrated rotameter or a dry gas meter must be used for measuring flow and volume. When sampling is complete, the tube should be capped with plastic caps; under no circumstances should rubber caps be used. At least one tube should be handled in the same manner as the sample tube (break, seal, and ship), except that no gas is passed through it. Label this tube as a blank.

6.2.2 Line Pressure Less Than 20 cm H_2O. Assemble the sampling apparatus (Figure A-1) with an empty sampling tube attached and connect it to the sampling port. (NOTE: The impinger is optional but its inclusion is recommended to keep

condensate from reaching the Florisil tube.) Open the port valve approximately one turn and observe the Magnehelic reading. If it is less than 20 cm H_2O, open the sampling system valve and set a sampling rate of 500 cc/min or less in the system by adjusting the port and sampling system valves. Now close off the sample port valve, noting the number of turns required. Substitute a Florisil-containing sampling tube for the empty one and open the sampling port valve until the desired flow rate is obtained. Record the time and either the initial volume (if a gas meter is being used) or the initial flow rate (if a rotameter is being used). (NOTE: If a rotameter is used, the flow should be monitored at 15 minute intervals.) When the required volume of gas has been sampled (minimum volume 50 liters), close off the sample port valve and remove the sampling system. Examine the Florisil tube to determine if pipeline condensate has contaminated it. If contamination has occurred, discard the sample and repeat the test. Otherwise seal the tube with plastic caps and prepare it for shipment to the laboratory for analysis. Record the sampling time and the volume of gas sampled. Discard the impinger contents.

6.2.3 Line Pressure Greater Than 20 cm H_2O. In this situation, a stainless steel micrometer valve with metal to metal seats must be attached to the sample port, and an orifice then attached to this valve to reduce the line pressure to less than 20 cm H_2O as required by the sampling procedure. If the pressure in the line is much larger than 20 cm H_2O, sufficient cooling may occur as the pressure is reduced to cause condensation in the micrometer valve and orifice. If this occurs, it can be corrected by heating the micrometer valve and orifice during sampling.

Once the pressure has been reduced to less than 20 cm H_2O, the sampling procedure is essentially the same as that in Section 6.2.2, except that the flow control valve downstream of the Florisil tube is optional.

6.3 Storage and Shipping of the Samples. Pack the tubes tightly before shipping to minimize breakage in transit. Avoid subjecting the tubes to high temperature or low pressure conditions. The samples should be stable for at least two months.

6.4 Analysis

6.4.1 Sample Preparation. Score the tube with a file, break it, and place its entire contents in an extraction column. (Alternately, the front and rear sections of the tube may be analyzed separately to check for break-through.) Add 10 ml hexane to the column and allow it to drain through the column into a separatory funnel at a rate of 1 to 2 ml/minute. Now add 10 ml concentrated sulfuric acid to the funnel and shake the mixture for 30 seconds. Remove the acid layer, discard it, and add 10 ml distilled, deionized water to the funnel and again shake for 30 seconds. Discard the water layer, pass the hexane layer through a sodium sulfate column into a 10 ml volumetric flask, and dilute to volume. Note, if a lower detection limit is desired, the hexane can be collected in a Kuderna-Danish ampoule and concentrated to a predetermined volume.

6.4.2 Analytical Procedure.

6.4.2.1 Preliminary Analysis. An injection of 2 µl sample is made when the conditions which will supply the necessary instrument response and separation occur. If the sample is too concentrated, dilutions must be made in order to bring the concentration into the linear or operating range of the electron capture detector. Once the concentration of the sample is appropriately adjusted, the chromatogram of this sample should be compared to that of a standard to determine if the pattern for the gas sample is qualitatively different from the Aroclor standards. If there is little difference in the two chromatograms, then proceed via the Standard

Analytical Procedure (6.4.2.2). If there is a significant difference in the two chromatograms, the Webb and McCall (6.4.2.3), or the perchlorination (6.4.2.4) procedure is followed, but only after a second column is used as added proof that polychlorinated biphenyls are likely present. Unless five or more peaks have retention times identical to PCB isomers on two columns, PCB is assumed to be absent in the sample.

6.4.2.2 Standard Analytical Procedure. An aliquot of up to 5 ul sample solution is injected. The syringe must be cleaned with hexane after each injection. The peak heights or areas of at least five prominent peaks in the sample chromatogram are selected and compared with those of the standard PCB mixtures, and the weight of PCB present is calculated.

6.4.2.3 The Webb-McCall Procedure. The concentration of PCB in the sample (ng/ml) is calculated using the area of the major peaks identified as PCB and the weight percent tables calculated by Webb and McCall. Care should be exercised to make certain that the relative order of peaks is the same as Webb and McCall's before using this procedure.

6.4.2.4 Perchlorination Procedure of Haile and Baladi. Pipette two 4.0 ml aliquots into culture tubes for perchlorination. Evaporate the aliquots in the culture tubes just to dryness with a gentle stream of dry nitrogen. To each tube add 0.2 ml antimony pentachloride with a 1-ml glass-TFE® syringe and seal the tube with a TFE®-lined screw cap. Heat the reaction mixture to 160°C for 2 hr by placing the tube in a hole in an aluminum block on a hot plate. Allow the tube to cool to room temperature before adding about 2 ml 50% HCl in water to destroy residual antimony pentachloride. This is a convenient "stopping point" in the perchlorination procedure.

Extract the reaction mixture. Add about 1 ml hexane to the tube, shake it, and allow the layers to separate. Remove the upper hexane layer

with a disposable pipette and filter through a micro-column of anhydrous sodium sulfate directly into a 5 ml volumetric flask. Repeat the extraction three times for a total of four extractions. Dilute the extract to volume with hexane. (Note, to serve as a control, a sample of decachlorobiphenyl (DCB) should also be carried through this procedure as should the blank tube.)

Analyze the perchlorinated extracts for DCB by gas chromatographic comparison with DCB standard solutions and correct this result for the DCB concentration determined for the blank tube.

7. Calculations

7.1 Standard Analytical Procedure. The following are the steps in the calculation of PCB concentrations determined with PCB mixtures as the standard.

7.1.1 The height or area of prominent peaks (minimum five) in the chromatogram are added, and compared to the total height or area of those same peaks in the Aroclor standards, and the concentration of the sample solution is read from the standard curve.

7.1.2 The concentration, of sample (ng/ml) solution is multiplied by the total volume of sample solution (ml) and the weight of PCB in the sample is calculated. Corrections for the blanks are made, if necessary.

7.1.3 The total weight of PCB in the gas sample is divided by the volume of gas sampled (liters) and the concentration is reported in ng/l or the equivalent units, micrograms per cubic meter.

7.2 The Webb-McCall Analytical Procedure. The following are the steps in the calculation of gas concentration of PCB determined by the method of Webb and McCall using Aroclor 1242 as an example.

7.2.1 The height or area of each peak in the chromatogram is compared to the corresponding Webb-McCall standard curve (Tables 1,2,3,4) and the concentration of particular PCB isomer(s) represented by that peak is read from the standard curve.

7.2.2 The concentration of each PCB is summed and multiplied by the total volume of sample solution (ml) to determine the weight of PCB in the sample. Corrections for blanks should be made, if necessary.

7.2.3 The total weight of PCB in the air sample is divided by the volume of air sampled (liters) and the concentration is reported in ng/l or the equivalent units, micrograms per cubic meter.

7.3 Perchlorination Procedure. The weight of DCB is calculated in a manner analogous to the standard analytical procedure (Section 7.1). The weight of DCB will be much larger than the weight of PCB present and this difference should be considered in reporting the results.

8. Calibration and Standards

8.1 GC Calibration. Aroclor standards (from 8 to 1000 ng/ml) are prepared in hexane by dilutions from a 1 mg/ml stock standard in hexane. A calibration curve should be established to check the linear range of the electron capture detector. The calibration curve should be checked daily by injection of at least two standards of different concentrations. If the perchlorination procedure is used, a standard of DCB in hexane is used for instrument calibration.

8.2 Flow Meter Calibration. Before use in the field, the accuracy of the dry gas meter or rotameter must be checked against a spirometer, a wet test meter, or any other suitable device. Natural gas must be used in calibrating rotameters, because gas viscosity affects rotameter calibration.

9. References

9.1 Webb, R. G. and A. C. McCall, J. Chromatogr. Sci. 11:366, 1973.

9.2 Haile, C. and Baladi, E. Methods for Determining the Polychlorinated Biphenyl Emissions from Incineration and Capacitor Filling Plants. EPA-600/4-77-048 U.S. Environmental Protection Agency Report. November 1977. NTIS No. PB-276-745/7BE.

9.3 Manual of Analytical Quality Control for Pesticides in Human and Environmental Media. EPA-600/1-79-008 U.S. Environmental Protection Agency Report.

TABLE 1. COMPOSITION OF AROCLOR 1242[a]

RRT[b]	Mean Weight (%)	Relative Std. Dev.[c]	No. of Chlorines[d]
11	1.1	35.7	1
16	2.9	4.2	2
21	11.2	3.0	2
28	11.0	5.0	2* (25%) 3* (75%)
32	6.1	4.7	3
37	11.5	5.7	3
40	11.1	6.2	3
47	8.8	4.3	4
54	6.8	2.9	3* (33%) 4* (67%)
58	5.6	3.3	4
70	10.3	2.8	4* (90%) 5* (10%)
78	3.6	4.2	4
84	2.7	9.7	5
98	1.5	9.4	5
104	2.3	16.4	5
125	1.6	20.4	5* (85%) 6* (15%)
146	1.0	19.9	5* (75%) 6* (25%)
Total	98.5		

[a]Taken from Reference 9.1.

[b]Retention time relative to p,p"-DDE = 100. Measured from first appearance of solvent, (3% OV-101 column).

[c]Standard deviation of six results as a percentage of the mean of the results.

[d]From GC-MS data. Peaks containing mixtures of isomers of different chlorine numbers have asterisks.

TABLE 2. COMPOSITION OF AROCLOR 1248[a]

RRT[b]	Mean Weight (%)	Relative Std. Dev.[c]	No. of Chlorines[d]
21	1.2	23.9	2
28	5.2	3.3	3
32	3.2	3.8	3
37	8.3	3.6	3
40	8.3	3.9	3* (85%) 4* (15%)
47	15.6	1.1	4*
54	9.7	6.0	3* (10%) 4* (90%)
58	9.3	5.8	4
70	19.0	1.4	4* (80%) 5* (20%)
78	6.6	2.7	4
84	4.9	2.6	5
98	3.2	3.2	5
104	3.3	3.6	4* (10%) 5* (90%)
112	1.2	6.6	5
125	2.6	5.9	5* (90%) 6* (10%)
146	1.5	10.0	5* (85%) 6* (15%)
Total	103.1		

[a]Taken from Reference 9.1.

[b]Retention time relative to p,p'-DDE - 100. Measured from first appearance of solvent, (3% OV-101 column).

[c]Standard deviation of six results as a percentage of the mean of the results.

[d]From GC-MS data. Peaks containing mixtures of isomers of different chlorine numbers have asterisks.

TABLE 3. COMPOSITION OF AROCLOR 1254[a]

RRT[b]	Mean Weight (%)	Relative Std. Dev.[c]	No. of Chlorines[d]
47	6.2	3.7	4
54	2.9	2.6	4
58	1.4	2.8	4
70	13.2	2.7	4* (25%) 5* (75%)
84	17.3	1.9	5
98	7.5	5.3	5
104	13.6	3.8	5
125	15.0	2.4	5* (70%) 6* (30%)
146	10.4	2.7	5* (30%) 6* (70%)
160	1.3	8.4	6
174	8.4	5.5	6
203	1.8	18.6	6
232	1.0	26.1	7

[a]Taken from Reference 9.1.

[b]Retention time relative to p,p'-DDE = 100. Measured from first appearance of solvent, (3% OV-101 column).

[c]Standard deviation of six results as a percentage of the mean of the results.

[d]From GC-MS data. Peaks containing mixtures of isomers of different chlorine numbers have asterisks.

TABLE 4. COMPOSITION OF AROCLOR 1260[a]

RRT[b]	Mean Weight (%)	Relative Std. Dev.[c]	No. of Chlorines[d]
70	2.7	6.3	5
84	4.7	1.6	5
98	3.8	3.5	e
104			5* (60%)
			6* (40%)
117	3.3	6.7	6
125	12.3	3.3	5* (15%)
			6* (85%)
146	14.1	3.6	6
160	4.9	2.2	6* (50%)
			7* (50%)
174	12.4	2.7	6
203	9.3	4.0	6* (10%)
			7* (90%)
232			f
244	9.8	3.4	6* (10%)
			7* (90%)
280	11.0	2.4	7
332	4.2	5.0	7
372	4.0	8.6	8
448	0.6	25.3	8
528	1.5	10.2	8
Total	98.6		

[a]Taken from Reference 9.1.

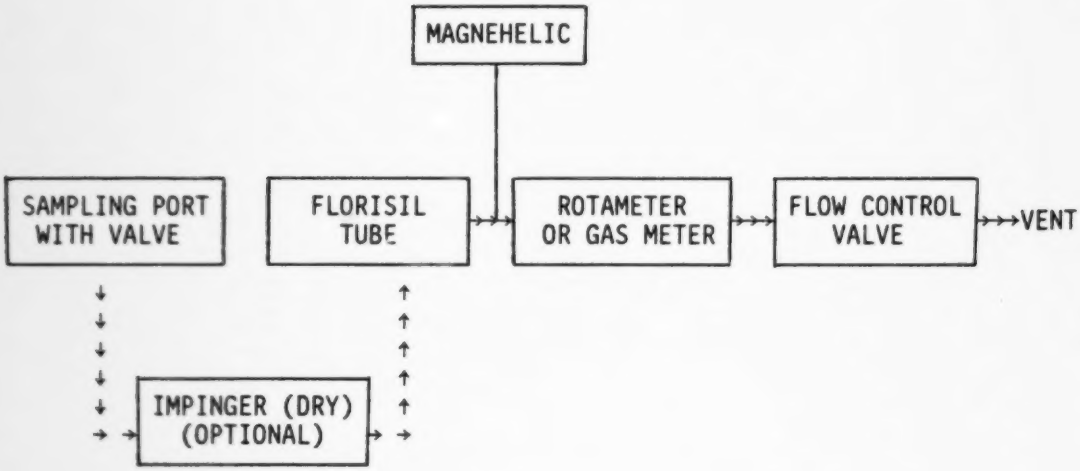
[b]Retention time relative to p,p'-DDE = 100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak have asterisks, (3% OV-101 column).

[c]Standard deviation of six results as a percentage of the mean of the results.

[d]From GC-MS data. Peaks containing mixtures of isomers of different chlorine numbers have asterisks.

[e]Composition determined at the center of peak 104.

[f]Composition determined at the center of peak 232.



(NOTE: Vent should be large enough not to cause a backpressure to occur in the system as this will affect rotameter accuracy)

Figure A-1. Schematic of sampling system